I.   ORTHO-SULPHAMINEBENZOIC ACID.

II.   ORTHO-CARBAMINEBENZENESULPHONIC ACID.

A Dissertation submitted to the Board of
University Studies of the Johns Hopkins Uni-
versity for the Degree of Doctor of Philosophy.

by

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Introduction.

ORTHOSULPHUMINEBENZOIC ACID.

Introduction.

Remsen and Fahlberg by oxidizing orthotoluene sul-
phamide with potassium permanganate discovered the sub-
stance known as benzoic sulphinide. W. A. Noyes by oxi-[1]
dizing ortho toluene sulphamide with potassium ferricyanide
discovered ortho sulphamine benzoic acid. He gave to the
acid the formula $C_6H_4 \begin{smallmatrix} COOH \\ SO_2NH_2 \end{smallmatrix}$. The acid is described as
being easily soluble in alcohol and ether and quite readily
soluble in cold water. It crystallized in two different
forms under different conditions: From the barium salt,
acidified with hydrochloric acid and allowed to cool, the
acid separated out in fine needles.

From the barium salt, acidified with hydrochloric
acid and allowed to stand in a cool place for several days,
the acid separated out in large clear prismatic crystals,
which appeared to belong to the monoclinic system.

Mr. Noyes also states that the acid, when heated
rapidly, melts at 165° to 167°. When heated slowly it
melts at $153^\circ - 155^\circ$. He thinks this is due to the fact

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(1)   Amm. Chem. Jour. 8 - 176.

that the acid is partially converted into benzoic sulphinide, for if the acid is kept at that temperature for a short while it becomes a solid, and then melts at 210° c. The substance in the tube, he says, has the intensely sweet taste which is so characteristic of benzoic sulphinide.

He obtained pure sulphinide by heating a portion of the acid to 180° c. in an air-bath. He thinks this change is due to a loss of water of constitution and not of crystallization, as evidenced by the fact that the salts of the acid are totally distinct from the salts of benzoic sulphinide.

PREPARATION OF ORTHO SULPHAMINE BENZOIC ACID.

Purification of Benzoic Sulphimide.

It occurred to Professor Remsen that the acid could be prepared by boiling pure benzoic sulphimide with sodium hydroxide. The benzoic sulphimide was purified by the method suggested by Pope, which consists in treating the (1) sulphimide with acetone. The sulphimide is more soluble in hot than in cold acetone. The para sulphamine benzoic acid, which is sometimes found in benzoic sulphinide as an
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(1) Jour. Chem. Society - 6 - 985.

impurity, is insoluble in acetone. This being the case, it is an easy matter to separate the two substances. The sulphinide used in the following experiments contained scarcely a trace of para sulphamine benzoic acid, even before it was treated with ~~the~~ acetone. One hundred grams of the sulphinide required about 460 grams of acetone.

When the benzoic sulphinide is dissolved in hot acetone a white crystalline mass is deposited immediately upon cooling, but if the sulphinide is dissolved in cold acetone and the solution is allowed to evaporate spontaneously massive, colorless rhombohedrons are deposited, some of which are several millimeters in length.

Hydrolysis of Benzoic Sulphinide.

After a number of experiments it was found that when the benzoic sulphinide is treated with sodium hydroxide, under the following conditions, the orthosulphamine benzoic acid was produced in nearly a quantitative yield: Twenty grams of pure benzoic sulphinide was added to a caustic soda solution, which was prepared by dissolving fifteen grams of sodium hydroxide in five hundred c.c. of water. This solution was evaporated on a water-bath until the sweet taste of the sulphinide could no longer be detected. The

The solution was allowed to cool and then filtered. The acid was precipitated by means of hydrochloric acid. If the solution is stirred vigorously, after the addition of hydrochloric acid, the acid is precipitated at once as a mass composed of fine crystals. If allowed to stand over night without disturbance large monoclinic crystals are deposited. After filtering, the acid was dissolved in alcohol from which solution it crystallized in large transparent rhombohedrons.

The acid when heated slowly melted at 152° c.; when heated rapidly it melted at 159° c. (uncorrected). It is quite soluble in alcohol and water. Some of the acid was placed in a small boat which was placed in a glass tube with a diameter of about 15 m.m. One end of the tube was connected with a bottle containing a small quantity of sulphuric acid. The other end was connected with a cylinder which contained calcium chloride. By connecting this apparatus with a filter pump, a current of dry air could be drawn through the tube. If a current of air flowing quite rapidly was allowed to pass through the tube, the acid was decomposed when heated to about 128° c. A white sublimate, which was very sweet, collected on the wall of the tube. If the current of air was shut off, the acid decomposed at

135^{0} , and white transparent needles of pure benzoic sulphi-
nide collected on the wall of the tube. Some of the need-
les were from ~~ten~~ to ~~twelve~~ m.m. in length. The acid con-
tains no water of crystallization.

The acid crystallizes in white opaque needles if the
solution is allowed to stand at least one week before crys-
tallization takes place. The needles melted at 152^{0}. The
needles are more soluble in water than the plates. The
needle acid contains water of crystallization. Below 110^{0}
it lost just one-half a molecule of water.

Calculated for	Found

$C_6 H_4 \diagdown \begin{matrix} COOH \\ So\ NH_2 \end{matrix}$ $- 1/2\ H_2O$ I.
$H_2O \quad 4.29$ 4.28

The needles when dissolved in alcohol crystallize
from the solution in plates which melt at 152 .

The needles decomposed when heated to about 115^{0}; a
white sublimate collected on the walls of the tube. The
sublimate was very sweet. Ten grams of benzoic sulphinide
was added to a caustic soda solution which was prepared by
dissolving twenty grams of caustic soda in 250c.c. of wa-
ter. The solution was treated in the same way as the pre-
vious solution. After treating the solution with hydrochlo-

acid if it is allowed to stand over night the acid crystallizes out in the form of needles. These proportions give nearly a quantitative yield of the needle acid.

A careful examination of the needles showed that they agreed in every respect with the needles obtained by the recrystallization of the plate acid from water. Melting point 152° ; if the temperature is kept at 152° for a while the acid becomes a solid which melts at 215°. If heated in the apparatus previously described the needles lose one-half molecule of water of crystallization below 100° c.

I - 0.3484 gram of acid lost 0.0150 gram of water at 90°.
II. 0.2920 gram of acid lost 0.0119 gram of water at 98°.

Calculated for   Found.

C_6H_4⟨COOH / SO$_2$NH$_2$⟩ - 1/2 H$_2$O   I.   II.

                                        4.30   4.11

        H$_2$O   4.24

If a current of air was allowed to flow rapidly through the tube, the acid decomposed at 115°c. and was partially fused. When the current of air was cut off en-

tirely, the acid decomposed at 145°. The acid was heated
at this temperature for about six hours. During this time
beautiful transparent needles of pure benzoic sulphinide,
some of which were several millimeters in length, collected
on the walls of the tubes. The needles were extremely
sweet and melted at 223° to 224° (uncorrected). By taking
proportions intermediate between the preparations used in
the previous experiment (fifteen grams of sodium hydroxide
with the same amount of benzoic sulphine acid water) a
yield is obtained which consists partly of the plate acid
and partly of the needle acid. On account of the differ-
ence in solubility they can be easily separated, the plates
being less soluble than the needles.

20 grams of benzoic sulphinide were added to a caus-
tic soda solution which was prepared by dissolving 20 grams
of sodium hydroxide in 750 c.c. water. The solution was
treated in the same way as in the previous experiments.
From 15 to 20 grams of acid were obtained. The solution
was set aside. After standing for a week or two, large
transparent, monoclinic plates were deposited, which were
fairly soluble in water. The crystals after being dried
became translucent. Upon examination the substance was
found to be a sodium salt, which, upon analysis, gave the

following result:

I.   0.1372 gram of the salt gave 0.0347 gram of Na_2SO_4
II. 0.1805 gram of the salt gave 0.0445 gram of Na_2SO_4
III. 0.1084 gram of the salt gave 0.0266 gram of Na_2SO_4

Calculated for	Found
Na	I. II. III.
	8.16 7.98 7.93

No loss in weight when heated to 165°, neither did it become sweet.

10 grams of benzoic sulphinide were added to a caustic potash solution which was prepared by dissolving 15 grams of potassium hydroxide in 250 c.c. of water. The solution was treated in the same manner as the previous solutions. After treating the solution with hydrochloric acid it was allowed to stand for several days. During this time large monoclinic plates were deposited. The plates became transparent when dried. Upon examination the substance turned out to be a potassium salt which, upon analysis, gave the following results:

I. 0.2149 gram of salt gave 0.0739 gram of K_2SO_4
II. 0.1478 gram of salt gave 0.0502 gram of K_2SO_4

Calculated for   Found

K   I.   II.

                                   15.40   15.22

No loss in weight when heated to 160°.

        20 grams of benzoic sulphinide were added to a caus-
tic soda solution which was prepared by dissolving 30 grams
of sodium hydroxide in 500c.c. of water. The solution was
treated in the same manner as the other solutions, with the
exception of placing the beaker in cold water when the hy-
drochloric acid was added. When the acid was added to the
solution quite a considerable quantity of heat was evolved.
After cooling, a crystalline mass was deposited. This mass
when crystallized from water separated as transparent, mo-
noclinic plates which remained transparent after being dri-
ed. The substance turned out to be the acid ammonium salt
of orthosulphobenzoic acid as evidenced by the fact that
ammonia could be detected when the salt was treated with a
solution of sodium hydroxide in the cold. Also by the fol-
lowing analysis:

Calculated for		Found.
COOH		I. II.
C_6H_4 SO$_2$ONH$_4$		38.74 38.79
C - 38.35		4.21 4.35
H 4.10		4.65
N 6.39		

The salt was converted into potassium salt which was ana-
lysed:

        K   16.25   16.36

Salts of Ortho-sulphaminebenzoic Acid.

          All of the salts crystallize from a water solution,
in well formed crystals. All of them contain water of
crystallization with the exception of the mono silver, po-
tassium and common salts. Without exception they all be-
come sweet when they lose their water of crystallization;
indeed they begin to get sweet just as soon as they begin
to lose their water.

          All the salts decompose at a few degrees above the
temperature at which they give off their water of crystal-
lization.

Sodium Ortho-sulphamine-benzoate -

$C_6H_4 \begin{array}{l} COONa \\ SO_2 NH_2 \end{array} + 2\frac{1}{2}H_2O$. This salt was prepared by treating a hot

solution of the acid with pure sodium carbonate to the point of neutralization. The salt crystallizes from an exceedingly concentrated water solution in fine silky needles. Only a few needles were obtained from a solution which was allowed to stand several weeks, in a small test tube. Even then a slight disturbance of the tube would cause the needles to redissolve. As a general rule the salt is deposited as a white amorphous mass. The salt becomes sweet by simply exposing it to the air for a short while. The salt was dried (with drying paper) and analysed at once. The percentage of sodium agreed with 1 1/2 molecules of water, and the percentage of water of crystallization agreed with 2 1/2 molecules of water.

I.   0.2114 gram of salt lost 0.0357 gram of water below 110°.

II.   0.2210 gram of salt lost 0.0375 gram of water below 110°.

III. 0.2628 gram of salt lost 0.0442 gram of water below 110°.

Calculated for   Found

$C_6H_4 \big\langle {\!\!\begin{array}{l} \text{COONa} \\ \text{SO}_2\text{NH}_2 \end{array}}$ - 2 1/2 H O   I.   II.   III.

 H₂O   16.79   16.88 16.96 16.81

Calculated for   Found

      COONa   I.   II   III

C_6H_4 + 1 1/2 H O

      SO_2NH_2 9.10 9.12 9.11

      Na   9.20

A few of the needles were obtained and an analysis

of the sodium agreed with 2 1/2 molecules of water.

I.   0.0174 grams of salt gave 0.0046 grams of Na_2SO_4.

II. 0.1945 grams of salt gave 0.0546 grams of Na_2SO_4.

Calculated for   Found

     COONa

C_6H_4 - 2 1/2 H O   I.   II.

     SO_2NH_2 8.62 8.58

     Na   8.58

Potassium Orthosulphamine benzoate.

     COOK

C_6H_4 This salt was prepared by neutralizing

     SO_2NH_2

the acid with pure potassium carbonate. The salt is ex-

tremely soluble in water, insoluble in methyl alcohol.

The salt is deposited from a water solution as a white amor-

phous mass. It crystallizes from a mixture of alcohol and

water in large transparent plates.

I. 0.1445 gram of salt gave 0.0528 gram of K_2SO_4.

II. 0.1265 gram of salt gave 0.0457 gram of K_2SO_4.

Calculated for		Found	
		I.	II.
C_6H_4 — COOK			
— SO_2NH_2		16.33	16.20
K 16.32			
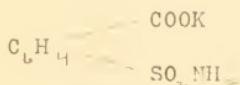
Calcium Orthosulphamine benzoate.

$(C_6H_4 \begin{array}{c} -COO \\ -SO_2NH_2 \end{array})_2$ Ca + $2H_2O$.   A hot solution of the acid was neutralized with pure calcium carbonate. The salt crystallized from a water solution in well formed translucent crystals.

If crystallization takes place at once, well formed crystals are obtained; but if the solution is allowed to stand several days, the salt is deposited as a white crystalline mass.

I. 0.5475 gram of salt lost 0.0414 gram of water below 140°.

II. 0.3776 gram of salt lost 0.0277 gram of water below 140°.

I.   0.2114 gram of salt gave 0.0598 gram of $CaSO_4$.

II. 0.2926 gram of salt gave 0.0829 gram of $CaSO_4$.

Calculated for   Found

$$(C_6H_4 {<COO \atop SO_2NH_2}) Ca - 2H_2O$$

	I	II
	8.27	8.30
Ca 8.40	7.56	7.33
H$_2$O 7.56		

Barium Orthosulphamine benzoate –

$$(C_6H_4 {<COO \atop SO_2NH_2})_2 Ba + 3H_2O -$$ This salt was prepared by
treating the acid with pure barium carbonate. It is quite
soluble in water and fairly soluble in alcohol. It crys-
tallizes from a water solution in transparent plates, and
sometimes it crystallizes from a water solution in needles
in the form of rosettes. It crystallizes in needles from
alcohol solution. Indications observed in different exper-
iments, point to the conclusion that the salt crystallizes,
under different conditions, with a different number of
molecules of water of crystallization. It agrees in every
respect with the salt prepared by Mr. W. A. Noyes [1] with the
exception of the number of molecules of water of crystalli-
zation. Mr. Noyes prepared two salts, one with 2 molecules
and another one with 9 molecules of water of crystalliza-
tion. This salt loses its water of crystallization when

approved etc. (?). aux.

0.1823 gram of the salt lost 0.0171 gram of H_2O below 150°.

I.   0.1214 gram of salt gave 0.0483 gram of Ba So_4

II. 0.1076 gram of salt gave 0.0426 gram of Ba SO_4

Calculated for		Found	
$(C_6H_4 \begin{matrix} COO \\ So_2NH_2 \end{matrix})_2 Ba + 3 H_2O$		I	II
Ba	23.18	23.39	23.32
H_2O	9.14	9.14	

exposed to the air.

Strontium Ortho sulphamine benzoate -

$(C_6H_4 \begin{matrix} COO \\ SO_2NH_2 \end{matrix})_2 Sr + 2H_2O$.   This salt is prepared by neu-
tralizing a hot solution of the acid with pure strontium

carbonate. If the solution is allowed to evaporate sponta-

neously the salt crystallizes in long transparent needle-

like crystals, some of which are three or four millimeters

in length. The crystals remain transparent when exposed to

the air.

I.   0.2151 gram of salt lost 0.0150 gram of H_2O below 140°.

I.   0.1385 gram of salt gave 0.0486 gram of Sr SO_4.

II.   0.2231 gram of salt gave 0.0783 gram of Sr.SO_4.

Calculated for $(C_6H_4 \begin{matrix} COO \\ SO_2NH_2 \end{matrix})_2 Sr + 2H_2O$.		Found	
		I	II
		16.71	16.67
Sr.	16.71		
		6.97	
H_2O	6.87		

Zinc Ortho sulphaminebenzoate -

$(C_6H_4 \begin{matrix} COO \\ SO_2NH_2 \end{matrix})_2 Zn - 3H_2O$. Two grams of the acid was dis-

solved in a sufficient quantity of water and pure zinc car-
bonate was added till the solution was neutral. The solu-
tion was evaporated to a slight extent, and allowed to
stand in a dessicator over night. Short opaque needles
-like crystals were deposited during the night. If the so-
lution is allowed to evaporate spontaneously, after sever-
al days, large transparent needle-like crystals are depos-
ited, some of which were from one to two centimeters in
length. The crystals do not lose their   transparency when
exposed to the air. The salt is fairly soluble in water.

I.   0.0809 gram of the salt lost 0.0081 gram of water be-
     low 130$^{\iota}$.

II.   0.0690 gram of salt lost 0.0070 gram of water below
      130$^{\iota}$.

I.   0.0449 gram of salt gave 0.0137 gram of $Zn\ SO_4$.

II.   0.0621 gram of salt gave 0.0192 gram of $Zn\ SO_4$.

III. 0.1476 gram of salt gave 0.0456 gram of $Zn\ SO_4$.

Calculated for   found.

$$\left(C_6H_4 \diagdown \begin{array}{c} COO \\ SO_3NH_4 \end{array} \diagup\right) Zn\ +\ 3H_2O$$
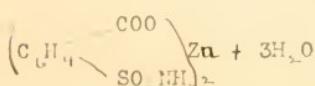
I	II	III
12.24	12.40	12.46
10.01	10.15	

         Zn   12.59
         H_2O 10.39

Zinc Ortho-sulphamine benzoate.-

$\left(C_6H_4\diagdown\begin{matrix}COO\\SO_2NH_2\end{matrix}\right)_2 Zn + 3H_2O.$ This salt was prepared from the needle acid in order to compare it with the zinc salt prepared from the plate acid. The two salts resemble one another in every respect with one exception. The solution of this salt, when allowed to evaporate spontaneously deposited large opaque needle-like crystals instead of transparent needles as is the case with the salt prepared from the plate acid.

I.   0.0652 gram of salt gave 0.0201 gram of $Zn SO_4$.

I.   0.0138 gram of salt lost 0.0138 gram of water below 150°.

Calculated for		Found
$\left(C_6H_4\diagdown\begin{matrix}COO\\SO_2NH_2\end{matrix}\right)_2 Zn + 3H_2O$		I.
		12.42
Zn 12.59		
		10.64
H_2O 10.39		

Magnesium Ortho sulphamine benzoate -

$\left(C_6H_4\diagdown\begin{matrix}COO\\SO_2NH\end{matrix}\right)$ $Mg + 4H_2O.$ - This salt was prepared by treating the acid with magnesium hydroxide. The salt

crystallized in well formed transparent needles which are deposited in the form of rosettes. The salt is extremely soluble in water.

I.   0.1850 gram of salt lost 0.0270 below 175°.
II. 0.2458 gram of salt lost 0.0355 gram of water below
       175°.

I.   0.2129 gram of salt gave 0.0519 gram of Mg SO_4.
II. 0.1726 gram of salt gave 0.0425 gram of Mg SO_4.
III. 0.1416 gram of salt gave 0.0341 gram of Mg SO_4.

Calculated for   Found

$\left(C_6 H_4 \diagdown \begin{array}{c} COO \\ SO_2 NH_2 \end{array} \right)_2 Mg + 4H_2O$

| | |
| I. II   III |
| 4.84 4.92 4.81 |

Mg   4.84
H_2O 14.52   14.59 14.42

Copper-Ortho sulphamine benzoate

$\left(C_6 H_4 \diagdown \begin{array}{c} COO \\ SO_2 NH_2 \end{array} \right)_2 Cu + 3H_2O$. - This salt was prepared by boiling a solution of the acid with freshly precipitated copper oxide, in a sand-bath, for about forty minutes. There was deposited after standing over night, large blue feathery needles, some of which were more than a centimeter in length. The salt is insoluble in alcohol and very diffi-

cultly soluble in water. The salt turned a deep green color when it lost its water of crystallization.

I. 0.1369 gram of the salt lost 0.0144 gram of water below 170°.

II. 0.0995 gram of salt lost 0.0095 gram of water below 170°

III. 0.0951 gram of salt lost 0.0085 gram of water below 170°.

I. 0.1684 gram of salt gave 0.0209 gram of Cu O.

II. 0.3018 gram of salt gave 0.0456 gram of Cu O.

Calculated for   Found

$$\left(C_6H_4 \begin{array}{l} - COO \\ \diagdown SO_2NH_2 \end{array} \right) Cu + 3H_2O$$

		I	II
		12.41	12.32
Cu	12.23		
		10.52	10.91
H_2O	10/44		

Mono Silver Ortho sulphamine benzoate. —

This salt was prepared by neutralizing two grams of acid with ammonium hydroxide and then boiling the solution until no odor of ammonia could be detected. Silver nitrate was added to the solution until no further precipitation

took place. By allowing the solution to stand over night there were deposited, fine feathery needles of a light gray color. The salt is soluble in hot water but insoluble in cold water. Becomes dark ~~colored~~ when exposed to the light.

Although it contains no water of crystallization it becomes sweet when heated up to 150°, and changes its color to light brown.

This salt resembles in every respect the salt pre-
(1)
pared by Mr. Noyes by the same method.

I.   0.0875 gram of salt gave 0.0307 gram of Ag.

II. 0.1171 gram of salt gave 0.0412 gram of Ag.

Calculated for		Found	
		I	II
C_6H_4 $\begin{cases} COOAg \\ SO_2NH_2 \end{cases}$		35.08	35.17
Ag 35.06			

Di-Silver Ortho-sulphamine-benzoate,

$C_6H \begin{cases} CO\ Ag \\ SO_2NHAg \end{cases} + H_2O$. — This salt was prepared by neutralizing about two grams of acid with ammonium hydroxide and then adding about 3/4 as much ammonium hydroxide in excess as was required to neutralize the solution. Silver nitrate

was added until no further precipitation took place. The salt was deposited as a white amorphous mass which was dissolved in boiling hot water. By allowing the solution to stand over night the salt separated out as silver-colored leaflets which became dark ~~colored~~ when exposed to the light. This salt is much less soluble in water than the mono silver salt, and resembles in every respect, with one exception, the di-silver salt prepared by Noyes [1] by treating the barium salt with silver nitrate. This salt contains one molecule of water of crystallization.

The salt was thoroughly dried and was allowed to remain wrapped in drying-paper for about one week. It was then heated to 140° in the apparatus previously described. The current of air was cut off, and water could be distinctly seen as it collected in drops in both ends of the tube. After losing its water it becomes a dark brown color.

I. 0.1231 gram of the salt lost 0.0053 gram of water at 167°.

I. 0.1867 gram of salt gave 0.0933 gram of Ag.

II. 0.1097 gram of salt gave 0.0549 gram of Ag.

[1] Loc. cit.

Calculated for

Found

	I	II
	49.97	50.04

C_6H_4 < COO Ag / SO$_2$ NHAg   + H$_2$O

Ag   49.88

H$_2$O   4.15

Ammonium Ortho-sulphamine-benzoate,

C_6H_4 < COO NH$_4$ / SO$_2$ NH$_2$ — This was prepared by neutralizing the acid with ammonium hydroxide. The salt is quite soluble in water, and crystallizes from a water solution in transparent plates.

The salt contains no water of crystallization, but becomes very sweet when heated to 125°, and decomposes quite easily. *Action of Phosphorus Oxychloride on the Sulphamine A...*

Five grams of the acid were mixed with five grams of phosphorus oxychloride and heated on a water-bath for two hours. No chloride was formed. All of the acid was converted into benzoic sulphimide.

$$C_6H_4 < \frac{CO\ OH}{SO_2\ NH\ H} \quad - \quad C_6H_4 < \frac{CO}{SO_2} > NH + H_2O.$$
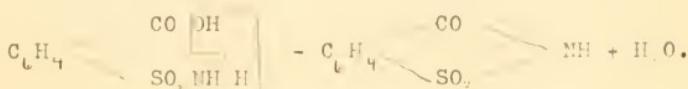
This reaction is evidence in favor of the formula given to

the acid by Dr. Noyes. In order to form benzoic sulphinide from the acid the hydroxyl group of the carboxyl group has to combine with one hydrogen atom of the amido group of the sulphone group as expressed by the above formula.

Preparation of the Diamide,

$C_6H_4 \begin{smallmatrix} CO\ NH_2 \\ SO_2\ NH_2 \end{smallmatrix}$.—The diamide was prepared by the following method:

Ten grams of the acid were mixed with six grams of ammonium cyanate. The mixture was heated, in a closed bath, for three days. During this time it was frequently stirred. As the acid is converted into benzoic sulphinide at quite a low temperature, the mixture was heated from $90°$ to $100°$ the first day, from $130°$ to $135°$ the second day and from $150°$ to $160°$ the third day. The mass, which is of a light straw color, is allowed to cool when it becomes brittle. It is then pulverized and treated with boiling alcohol in which it is soluble. After filtering, the solution is allowed to cool when there is deposited light colored needles. Melting-point $263°$.

$$C_6H_4 \begin{smallmatrix} COOH \\ SO_2\ NH_2 \end{smallmatrix} \quad \rightarrow \quad C_6H_4 \begin{smallmatrix} CO\ NH_2 \\ SO_2\ NH \end{smallmatrix}$$
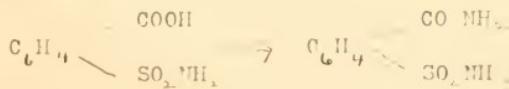
Analysis of the diamide gave the following results:

I.   0.1296 gram of diamide gave 0.1528 gram of Ba SO$_4$.

I.   0.1565 gram of the diamide gave 0.0209 gram of nitro-
gen.

II. 0.1413 gram of the diamide gave 0.0188 gram of nitro
gen.

Calculated for		Found	
	CO NH$_2$	I.	II.
C$_6$H$_4$ SO. NH$_2$		16.20	
S 16.00		13.35	13.27
N 14.00			

I am indebted to Mr. C. E. Waters for the nitrogen
determinations. Mr. Waters observed the fact that the
ammonia was given off in two different stages. One portion
of the ammonia was given off when the sodium hydroxide
solution was dilute; after heating sometime the second
portion was given off when the soda solution had become
very concentrated. So there must be some difference in
the stability of the two amide groups.

Part II.

ORTHOCARBAMINEBENZENE SULPHONIC ACID.

Introduction.

(1)

This acid was first prepared by Fahlberg and B
by the following method: Ortho sulphobenzoic acid when
treated with ammonia, reacts according to this equation:

$$C_6H_4 \begin{array}{c} CO \\ \diagdown \\ SO_2 \end{array} O + 2NH_3 = C_6H_4 \begin{array}{c} CO \ NH_2 \\ \diagdown \\ SO_2 \ ONH_4 \end{array}$$
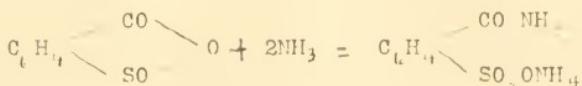
The ammonium salt when treated with silver nitrate
is asily converted into the silver salt. The free acid
is obtained by treating the silver salt with sulphuric
acid.

They describe the acid as crystallizing in thick
prismatic crystals which are easily soluble in water and
alcohol, and contain one molecule of water of crystalliza-
tion. Melting point 185 to 186 . (uncorrected). By melt-
ing the acid with potassium hydroxide, ammonia is given off,
but when simply heated no ammonia is given off.

(1)

The acid was prepared later by Jeansurun by heat-
ing orthocyanebenzene sulphone chloride with water or alco-


(1) Ber. d. Chem. Ges. 22, 7598
(2) Ber. d. Chem. Ges. 26, 2298.

hol.

(1)

Dr. Karslake   also obtained the acid by hydrolysis
of ortho-cyanbenzenesulphonic acid by means of dilute al-
kali:

$$C_6H_4 \begin{array}{c} CN \\ \diagdown \\ SO_2OH \end{array} + H_2O = C_6H_4 \begin{array}{c} CO\ NH_2 \\ \diagdown \\ SO_2OH \end{array}.$$
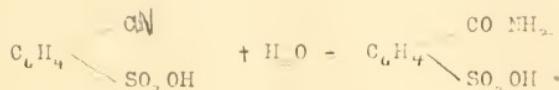
Preparation of Acid

The potassium salt of the acid can be obtained in any
desired quantity by treating the acid potassium salt of or-
tho_sulphobenzoic acid with ammonium sulphocyanate (2)

The acid potassium salt was prepared according to
(3)
the method of Remsen and Saunders.

A modification of the proportions suggested by their
method was made for the reason that the benzoic sulphinide
used, contained scarcely a trace of para sulphamine benzoic
acid.

The substances were mixed together in the proportion
of five parts of acid potassium salt to three parts of am-
monium sulphocyanate. The mixture was heated in a closed

bath for three days at a temperature from 180° to $200^{o} w$.
During this time it was occasionally stirred. The mass
which is of a brown color, was allowed to cool when it be-
comes a stiff cake. It is then finely pulverized and
digested with boiling alcohol several times. The potassium
salt of the acid which is in the form of sandy crystals was
separated from the alcohol by filtering. The salt when re-
crystallized from water separates out in large transparent
plates.

When the potassium salt is treated with silver ni-
trate, in the proportion of their molecular weights, reaction
takes place with the greatest ease with the formation of
the silver salt of ortho carbamine benzene sulphonic acid.
The acid is obtained by treating the silver salt with hy-
drochloric acid. The acid crystallizes from a very concen-
trated water solution in large, thick, transparent crystals
which are quite soluble in alcohol. Melting-point 193°-
$194^{o} w$ (uncorrected). The crystals remain transparent when
exposed to the air. It does not become sweet when it loses
its one molecule of water of crystallization, neither does
it become sweet by continued heating. It decomposes when
heated to 125^{u}.

I. 0.2691 gram of the acid lost 0.0228 gram of water be-
    low 120°.

II. 0.2585 gram of the acid lost 0.0214 gram of water be-
    low 120°.

         Calculated for   Found

                    CO NH₂   I.   II.
    C_6H_4 -   + H₂O
                    SO₂OH   8.47   8.27

              H₂O   8.22

Salts of Ortho-carbamine benzenesulphonic acid -

        The salts of this acid do not crystallize as well as
the salts of ortho-sulphamine benzoic acid, but are much
more soluble in water.

        Most of the salts contain water of crystallization
but none of them become sweet when they lose their water;
neither do they become sweet when they are decomposed.

Potassium ortho-carbamine benzene sulphonate

                    CONH₂
    C_6H_4 + H₂O . - This salt was prepared by
                    SO₂OK
by the method described in the preparation of the acid. It
crystallizes from a water solution in large transparent

plates. It is quite soluble in water and alcohol. It
crystallizes from alcohol in needles. The salt is deliques-
cent. If the large transparent crystals are placed in a
beaker and covered over with water, the moment they become
warm a crackling sound can be distinctly heard some dis-
tance from the water-bath. The crystals become opaque and
crumble to pieces. This phenomenon is doubtless due to the
fact that the salt loses its water of crystallization very
easily; in this instance $_\wedge$loses it under water.

probably
(1)

A similar salt is described by Karslake   derived
(2)
from cyanbenzenesulphonic acid, and by Sohon   derived
from the ammonium salt of benzaminesulphonic acid.

I.   0.3439 gram of salt lost 0.0239 gram of water below
      100 .
II.   0.5499 gram of salt lost 0.0380 gram of water below
      100 .
III. 0.4801 gram of salt lost 0.0331 gram of water below
      100 .

I.   0.4660 gram of salt gave 0.1583 gram of K_2SO_4
II.   0.4904 gram of salt gave 0.1657 gram of K_2SO_4.
III. 0.4246 gram of salt gave 0.1441 gram of K_2SO_4.

----

(1) Dissertation, J.H.U., 1895, p.17
         "   "   1896, p.18

Calculated for			Found		
			I.	II.	III.
C_6H_4 $\begin{array}{c} CO\ NH \\ SO_2 OK \end{array}$ + H_2O			15.21	15.11	15.21
	K	15.21	6.94	6.91	6.84
	H_2O	7.00			

Silver Ortho-carbaminebenzene-sulphonate,

C_6H_4 $\begin{array}{c} CO\ NH_2 \\ SO_2 OAg \end{array}$ + H_2O . — When the potassium salt of
the carbamine acid is treated with silver nitrate, in the
proportion of their molecular weights, reaction takes place
with the greatest ease with the formation of the silver
salt. The salt which is fairly soluble in water crystal-
lizes in large monoclinic transparent plates. It is decom-
posed when brought in contact with wet filter paper, and be-
comes dark ~~colored~~ when exposed to the light for any length
of time. The salt is decomposed with the greatest ease
when treated with hydrochloric acid or with the chlorides
of the metals.

This salt resembles in every respect the salt pre-
pared by Fahlberg and Barge [1] by treating the ammonium salt
of the carbamine acid with silver nitrate.

----

(1) Berichte der D.C. 22, 759.

I.   0.3557 gram of salt lost 0.0193 gram of water below
     136 .

II. 0.4136 gram of salt lost 0.0228 gram of water below
     136 .

I.   0.2362 gram of salt gave 0.0794 gram of Ag.

II. 0.3681 gram of salt gave 0.1215 gram of Ag.

Calculated for   Found.

                 CO NH₂   I   II
C_6H_4 $\underset{\text{SO}_2\text{OAg}}{\overset{\text{CO NH}_2}{\diagdown}}$ + H₂O.

                                             33.19   33.00

            Ag   33.14   5.45   5.51
            H₂O   5.52

Tri-Silver Ortho-Carbaminebenzene-sulphonate

$C_6H_4 \underset{\text{SO}_2\text{OAg}}{\overset{\text{CONAg}_2}{\diagup}}$. — A small quantity of the potassium
                                    carbaminebenzenesulphonate was dissolved

in water and about two drops of ammonia were added to the

solution. A hot solution of silver nitrate was added until

no further precipitate was formed. The salt was deposited

in a white amorphous mass which was insoluble in boiling

water. The salt became dark ~~colored~~ when exposed to the

light and is extremely stable. It was with the greatest
difficulty that it was decomposed, therefore, the following
analysis cannot be reported with confidence.

Calculated for   Found.

C_6H_4 ⟨ CO NAg.   I

      ⟨ SO₂OAg   62.56

      Ag   62.07

Sodium Ortho-carbamine-benzene-sulphonate.

C_6H_4 ⟨ CONH₂ · SO₂ONa + 2H₂O. — This salt was prepared by treat-
                    ing the silver salt with sodium chloride.
It is quite soluble in water, from which solution it crys-
tallizes in large transparent plates which belong to the
monoclinic system.

        This resembles in every respect the salt described
        (1)
by Karslake.

I.   0.1771 gram of the salt lost 0.0244 gram of water be-
      low 140′.

II.   0.1572 gram of the salt lost 0.0217 gram of water be-
      low 145°.

Calculated for   Found

$$\text{C}_6\text{H} \underset{\displaystyle \text{SO}_2\text{ONa}}{\overset{\displaystyle \text{CONH}_2}{\diagdown}} \quad + 2\text{H}_2\text{O}.$$

	I.	II.
	8.85	8.80

Na   8.88   13.89   13.80

H$_2$O   13.85

Barium Ortho-carbamine-benzene-sulphonate.-

$$\text{C}_6\text{H}_4 \underset{\displaystyle \text{SO}_2\text{O}}{\overset{\displaystyle \text{CONH}_2}{\diagdown}} \Bigg\} \text{Ba} + 2\text{H}_2\text{O}.$$ — This salt was prepared by treat-
ing the silver salt with hydrochloric acid, and then adding
pure barium carbonate to the point of neutralization. The
salt crystallized in transparent plates from a very concen-
trated water solution.

The percentage of water of crystallization agrees
with two molecules of water. The percentage of barium
agrees with 2 1/2 molecules of water. The following anal-
yses were made of different portions of the salt prepared
at different times.

I.   0.1787 gram of salt lost 0.0114 gram of water below
      175°.

II.   0.2194 gram of salt lost 0.0138 gram of water below
      175°.

I.   0.0587 gram of salt gave 0.0357 gram of Ba SO$_4$.

II. 0.1584 gram of salt gave 0.0037 gram of Ba SO$_4$.

Calculated for   Found.

$$\left(C_6H_4 \left\langle \begin{array}{l} CONH_2 \\ SO_2O \end{array} \right. \right)_2 \quad Ba + 2H_2O$$

	I	II
	6.37	6.38

H$_2$O   6.38

Calculated for   Found.

$$\left(C_6H_4 \left\langle \begin{array}{l} CO\ NH_2 \\ SO_2O \end{array} \right. \right)_2 \quad Ba + 2\ 1/2\ H_2O$$

	I	II
	23.56	23.61

Ba   23.54

Strontium Ortho-carbaminebenzenesulphonate,

$$\left(C_6H_4 \left\langle \begin{array}{l} CO\ NH_2 \\ SO_2O \end{array} \right. \right)_2 Sr.$$ This salt was prepared just as the corresponding ba-

rium salt by using strontium carbonate. The salt separated,

out of an extremely concentrated water solution, as small,

transparent crystals with no definite form.

I.   0.2145 gram of the salt gave 0.0813 gram of Sr SO$_4$.

II. 0.2017 gram of the salt gave 0.0764 gram of Sr SO$_4$.

Calculated for   Found

$$\left(C_6H_4 \left\langle \begin{array}{l} CONH_2 \\ SO_2O \end{array} \right. \right)_2 Sr.$$

	18.08	18.04

Sr   17.85

Calcium Ortho-carbamine-benzene-sulphonate

$\left(C_6H_4 \begin{array}{c} \text{-CONH}_2 \\ \text{-SO}_2O \end{array} \right)_2$ Ca + 2H$_2$O. — This salt was prepared just as the corresponding strontium salt by using pure calcium carbonate. The salt crystallized in large transparent plates from a very concentrated water solution.

I. 0.1333 gram of salt lost 0.0097 gram of water below 185°.

II. 0.1986 gram of salt lost 0.0150 gram of water below 185°.

I. 0.1169 gram of salt gave 0.0332 gram of CaSO$_4$.

II. 0.1127 gram of salt gave 0.0323 gram of CaSO$_4$.

Calculated for		Found	
		I	II
$(C_6H_4 \begin{array}{c} \text{-CONH}_2 \\ \text{-SO}_2O \end{array})_2$ Ca + 2H$_2$O		7.28	7.95
Ca 7.56		8.39	8.43
H$_2$O 8.40			
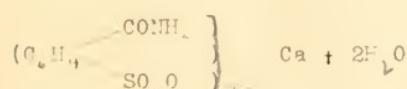
Zinc Ortho-carbamine-benzene-sulphonate

$(C_6H_4 \begin{array}{c} \text{-CONH}_2 \\ \text{-SO}_2O \end{array})$ Zn + 3H$_2$O . — This salt was prepared by treating the acid with pure zinc carbonate. The salt is

deposited from a very concentrated water solution in the
form of short thick needles. It is efflorescent.

I.   0.2187 gram of salt lost 0.0227 gram of water below
      150°.
II.   0.2477 gram of salt lost 0.0256 gram of H_2O below 150°

I.   0.0934 gram of salt gave 0.0286 gram of $ZnSO_4$.
II.   0.1022 gram of salt gave 0.0320 gram of $ZnSO_4$.

Calculated for		Found	
		I	II
$(C_6H_4 \begin{array}{l} CONH_2 \\ SO_2O \end{array}) Zn + 3H_2O.$		12.31	12.62
Zn 12.59		10.37	10.34
H_2O 10.39			

Magnesium carbamine benzene sulphonate ,

$(C_6H_4 \begin{array}{l} CONH_2 \\ SO_2O \end{array}) Mg + 2H_2O.$ – This salt was prepared just as
the zinc salt by using pure magnesium carbonate. The salt
separates out as a crystalline mass from a very concentra-
ted water solution.

I.   0.1983 gram of salt lost 0.0146 gram of water below
     140°.

II.   0.2002 gram of salt lost 0.0158 gram of water below
      140°.

I.   0.1204 gram of salt gave 0.0310 gram of $MgSO_4$.

II.   0.1904 gram of salt gave 0.0490 gram of $MgSO_4$.

Calculated for			Found	
			I	II
$(C_6H_4\genfrac{}{}{0pt}{}{CONH_2}{SO_2O})$	$Mg + 2H_2O$		5.14	5.14
			7.36	7.39
Mg	5.22			
H_2O	7.82			

Copper Ortho-carbamino-benzene sulphonate

$(C_6H_4\genfrac{}{}{0pt}{}{CONH_2}{SO_2O})$ $Cu + 4H_2O$. — This salt was prepared by
boiling the acid with copper oxide on a water-bath. When
allowed to cool, the salt was deposited as beautiful deep
blue plates. When the salt loses its water of crystalliza-
tion it becomes gray ~~colored~~. If a small quantity of water
is added to the gray ~~colored~~ salt it becomes light blue
~~colored~~.

I.   0.1761 gram of salt lost 0.0240 gram of water below
      200°.

II.   0.1760 gram of salt lost 0.0240 gram of water below
      200°.

I.   0.1083 gram of salt gave 0.0127 gram of CuO.

II.   0.0435 gram of salt gave 0.0064 gram of CuO.

Calculated for			Found	
			I	I
$(C_6H_4\begin{smallmatrix}CONH_2\\SO_2O\end{smallmatrix})_2$	$Cu + 4H_2O$		11.72	11.72
Cu 11.86			13.62	13.63
H_2O 13.45				

Ammonium Ortho-carbamine-benzene-sulphonate ¬

$C_6H_4\begin{smallmatrix}CONH_2\\SO_2ONH_4\end{smallmatrix}$, —   This salt was prepared by neutral-
izing the acid with ammonium hydroxide. It crystallized
from a concentrated water solution in large plates. One
plate obtained was at least 2 1/2 c.c. in length. It gives
off ammonia when treated with sodium hydroxide in the cold.
After heating the salt up to 140° no sweet taste could be

detected. Melts at 262° to 263°(uncorrected). If the current of air was cut off water collected in the ends of the tube when the salt was heated to 125°.

I. 0.1503 gram of salt lost 0.0062 gram of water below 125°.

II. 0.1404 gram of salt lost 0.0059 gram of water below 125°.

I. 0.1562 gram of salt gave 0.1649 gram of $Ba\ So_4$.

Calculated for		Found	
		I.	II
$C_6H_4 \diagdown \begin{array}{c} CONH_2 \\ SO_2ONH_4 \end{array} + 1/2\ H_2O$.		4.12	4.20
H_2O 3.96			

Calculated for	Found
	I
$C_6H_4 \diagdown \begin{array}{c} CONH_2 \\ SO\ ONH_4 \end{array}$	14.46
S. 14.67	

This salt was prepared by Fahlberg and Barge[1] and described as crystallizing from alcohol in white needles which melt at 225° - 226°. Jemsurun[2] describes it as crystal-

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(1) Loc. cit.   (2) Loc. cit.   (3 Loc. cit.

lizing from water in granular crystals containing one mole-
cule of water of crystallization. Karslake [1] describes the
salt as crystallizing from a water solution, in transparent
needles, which contain no water of crystallization and
which melted at 219° - 220° (uncorrected).

Preparation of Chloride.

Five grams of the potassium salt was mixed with five
grams of phosphorus oxychloride in a small flask. The
flask was attached to a return condenser, and then placed
in a bath which contained boiling water. The mixture was
heated for about one-half an hour. The white mass was pul-
verized in a mortar and treated with cold water, the ortho-
carbamine-benzene-sulphone-chloride, which remains in the
form of white crystalline powder insoluble in water, is fil-
tered off. It was then thoroughly dried. It crystallized
from chloroform solution in small thick opaque plates.
Melting-point 63 .

$$C_6 H_4 \begin{cases} CONH_2 \\ SO_2 OK \end{cases} \qquad C_6 H_4 \begin{cases} CONH \\ SO_2 Cl \end{cases}$$

I. 0.1156 gram of salt gave 0.0984 gram of AgCl (Carius
method).

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(1) Loc. cit.
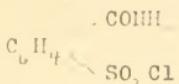
Calculated for   Found
   . COHH
C$_6$H$_4$ I.
      SO$_2$Cl   16.00

      Cl   16.17

Potassium Orthocyanbenzene-sulphonate

        CN
C$_6$H$_4$. _ This salt was prepared by treating the
      SO$_2$OK
potassium salt with phosphorus oxychloride as in the pre-
vious experiment, only the mixture in this experiment was
simply warmed in the water-bath. The whole of the potas-
sium salt was converted into the cyan salt. It crystal-
lizes from a moderately dilute water solution in small
transparent needles radiating from a common centre.

        If the mixture of the potassium salt and phosphorus
oxychloride is placed in a cold water bath, and then heated
to the boiling temperature, the potassium salt will be part-
ly converted into the chloride and partly into the cyan po-
tassium salt.

        CO NH$_2$ CN
C$_6$H$_4$ >   C$_6$H$_4$
        SO$_2$OK   SO$_2$OK
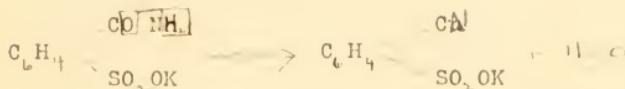
        This reaction is evidence in favor of the formula
given to the acid by Fahlberg and Barge.

This salt resembles in every respect the salt describ-
ed by Noyes, derived from ammonium cyanbenzenesulphonate.

(1)

Calculated for   Found.

$$C_6H_4 \begin{array}{l} - CN \\ - SO_2OK \end{array}$$

K   17.67

|	I	II
	17.67	17.71

A small portion of the chloride was placed in a
beaker which was then placed in a dish of cold water. The
chloride was treated with a small quantity of an ordinary
solution of ammonium hydroxide. The beaker was allowed to
stand in the water for about three hours, being stirred at
frequent intervals. It was then warmed on a water-bath
for about half an hour. The solution was then filtered and
a white crystalline substance remained on the filter paper.
It was soluble only in boiling alcohol from which solution
it crystallized in small opaque monoclinic (?) plates. The
substance did not melt when heated to the boiling-point of
sulphuric acid. A sulphur determination was made by the
Liebig method. Ammonia was given off when the substance
was fused with potassium hydroxide, although not as freely
as in the case of the sulphamine diamide. This substance
seemed to be more stable than the sulphamine diamide.

I.   0.1203 gram of substance gave 0.1506 gram of Ba SO$_4$.

Calculated for   Found

$C_6H_4\begin{cases} CO\ NH_2 \\ SO_2\ NH_2 \end{cases}$ I

S.   16   17.20

     Possibly the substance was not thoroughly oxidized
when fused with potassium hydroxide or else it is not the
carbamine diamide.

Appendix.

The following work was done by Mr. Benjamin Palmer
Caldwell, a student in the department of Physical Chemis-
try -

Determination of the Molecular Conductivity and Activity
Coefficient (Dissociation Constant) of Ortho-Sulphamine-
Benzoic Acid, $C_6H_4 \overset{COOH}{\underset{SO_2NH_2}{<}}$, and Ortho-Carbamine Sulpho-
Benzoic Acid,---------------- $C_6H_4 \overset{CONH_2}{\underset{SO_2OH}{<}}$.

benzene sulphonic
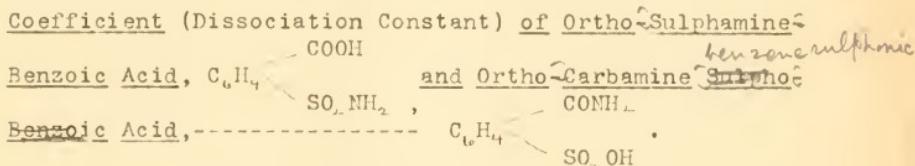
The conductivity was determined by the method of
Kohlrausch (Zeit. phys. Chem., I, 105; 2, 902) using a
Wheatstone bridge, induction coil, and telephone.

Since organic acids, in general, are not strongly
dissociated in solution, complete dissociation is only
reached at dilutions far too great to permit the use of the
conductivity method. The conductivity of the sodium salt
at infinite dilution is, therefore, obtained, since this is
strongly dissociated, and that of the acid is computed from
this value by subtracting the constant for the Sodium ion,
49, and adding to the remainder the constant for the Hydro-
gen ion, 325. In the case of the Ortho-sulphamine Benzoic
acid, this was not done, since the composition of the dry

sodium salt was not positively ascertained. The value 360,
was taken as the molecular conductivity of this acid at
complete dissociation, it having been seen in the examina-
tion of the results of Ostwald's work on organic acids
(Zeit.Phys.Chem. 3, 170,240,360.) that the value for a very
large number of closely related monobasic acids fell be-
tween 357 and 362. This number, for monobasic acids is the
sum of the Hydrogen constant, 325, and the constant for the
union made up of the rest of the acid. This latter constant
is nearly the same throughout, for a large number of acids.
The value, 360, cannot be wrong by more than two units -
which would not ~~naturally~~ materially affect the results dependent upon
it ~~entirely~~.

In the case of the Ortho-carbamine ~~Sulpho Benzoic~~ benzene sulphonic
acid, the sodium salt was employed, and the value for the
molecular conductivity of that acid at complete dissocia-
tion was computed as shown above.

The amount of dissociation at any specified dilu-
tion, α , is obtained by dividing the molecular conductivi-
ty at that dilution u_v, by the molecular conductivity at
infinite dilution u_∞ - that is, at complete dissoci-
ation: $\alpha = \dfrac{u_v}{u_\infty}$.

The chemical activity of acids is directly propor-

tional to the dissociation constant, $\underline{k}$. This is obtained

by the formula of Ostwald (Zeit.Phys. Chem., 3, 172), which

was adduced from the condition of a dissociating vapor

$$k = \frac{c \, x^2}{(1-x)v}$$

Where $\underline{V}$ is the number of litres of

solution containing a gram molecule of the dissolved sub-

stance. This formula holds for weakly dissociated substan-

ces, but fails when applied to strongly dissociated com-

pounds.

The results obtained by Mr. Caldwell are given be-

low with the acids, submitted to me for examination.

C_6H_4 $\begin{array}{l}\text{COOH} \\ \text{SO}_2\text{NH}_2\end{array}$		Molec. Wb.201 146	Temperature 25°
Volume	Conduc-tivity,	Dissociation,100	Dissociation con-stant, 100 k.
25	75.95	21.09	0.2254
50	101.74	28.23	0.2221
100	133.89	37.19	0.2361
200	171.05	47.65	0.2264
400	212.48	59.02	0.2125
800	251.10	69.07	0.1928
1600	278.12	77.02	-
3200	300.14	83.37	-
6400	323.41	89.83	-

12800   334.21   92.83   -

$\mathcal{U}_\infty$ = 360   100k (mean of first five) 0.2245.

I omit the last four values for the dissociation
constant. The conductivity of the water at high dilutions
brings in a slight error in the molecular conductivity as
determined, and slight errors there are often magnified ten
times or more in the calculation of dissociation constants.

$C_6H \underset{SO_2ONa}{\overset{CONH}{\diagdown}} + 2H_2O$ Molec. Wt. 259 213.   Temperature, 25°
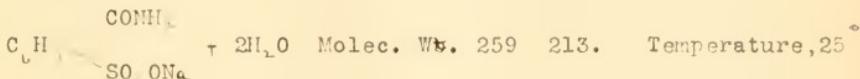
Volume	Conductivity
20	69.056
40	72.805
80	75.310
160	78.530
320	80.610
640	82.352
1280	85.506
2560	87.140

The error from the water commences to show itself at
N/1280 dilution. The correct conductivity for complete
dissociation is about 85.

85 - 49 + 325 = 361, u... of the acid.

C_6H_4 $\begin{array}{c} CONH \\ SO_3OH \end{array}$ + H_2O.   Molec. lot. 219 152.

u ... = 361.   Temperature 25
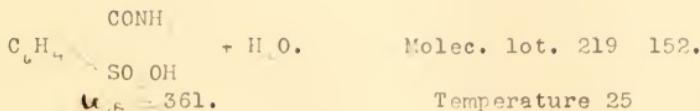
Volume.	Conductivity,	Dissociation, 100
50	317.01	87.81
100	326.53	90.45
200	331.96	91.95
400	334.65	92.70
800	334.68	92.70
1600	336.52	93.21
3200	344.70	95.48
6400	357.26	99.02
12800	Too dilute to work.	

100 k = 12.64.

The water used in all of the above work was distilled from acid potassium bichromate and alkaline potassium permanganate in the way described by Jones and Mackay in (Am.Chem.Jour., 19,91). Zeitsche Phys. Chem. 22,237.

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Biographical.

Francis Daniel Wilson was born in Chatham, Virginia, December 3, 1871. His primary education was received in the public school of that town. After engaging in the mercantile business for three years he entered the Virginia Polytechnic Institute at Blacksburg, Virginia in fall of 1890, and received the degree of Bachelor of Science in 1894, and Master of Science in 1896. During the two years, from 1894 to 1896, he was assistant in General and Analytical Chemistry. In the fall of 1896 he entered the Johns Hopkins as a student in Chemistry, Geology and Mineralogy.

Each year he was appointed to ordinary Virginia scholarships.